Cryogenic Distillation Plant
Rev. 5

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Introduction

The report presents the design of a cryogenic distillation system capable of purifying a gas stream composed by 99% argon and 1% nitrogen (percentage by volume). The purpose is to treat the crude argon stream from underground wells, extracted and refined with the VPSA system and the charcoal traps in Cortez, CO. The goal is to reduce the nitrogen contamination below 1 ppm. The residual nitrogen contamination at the output of the cryogenic distillation system will be removed with hot getters.

Figure 1 shows the vapor pressure curves of argon and nitrogen, and Table 1 shows the boiling points of the two compounds in the gas mixture: argon and nitrogen.

The different boiling points of the two compounds address the possibility to perform a cryogenic distillation to remove nitrogen from the gas mixture.

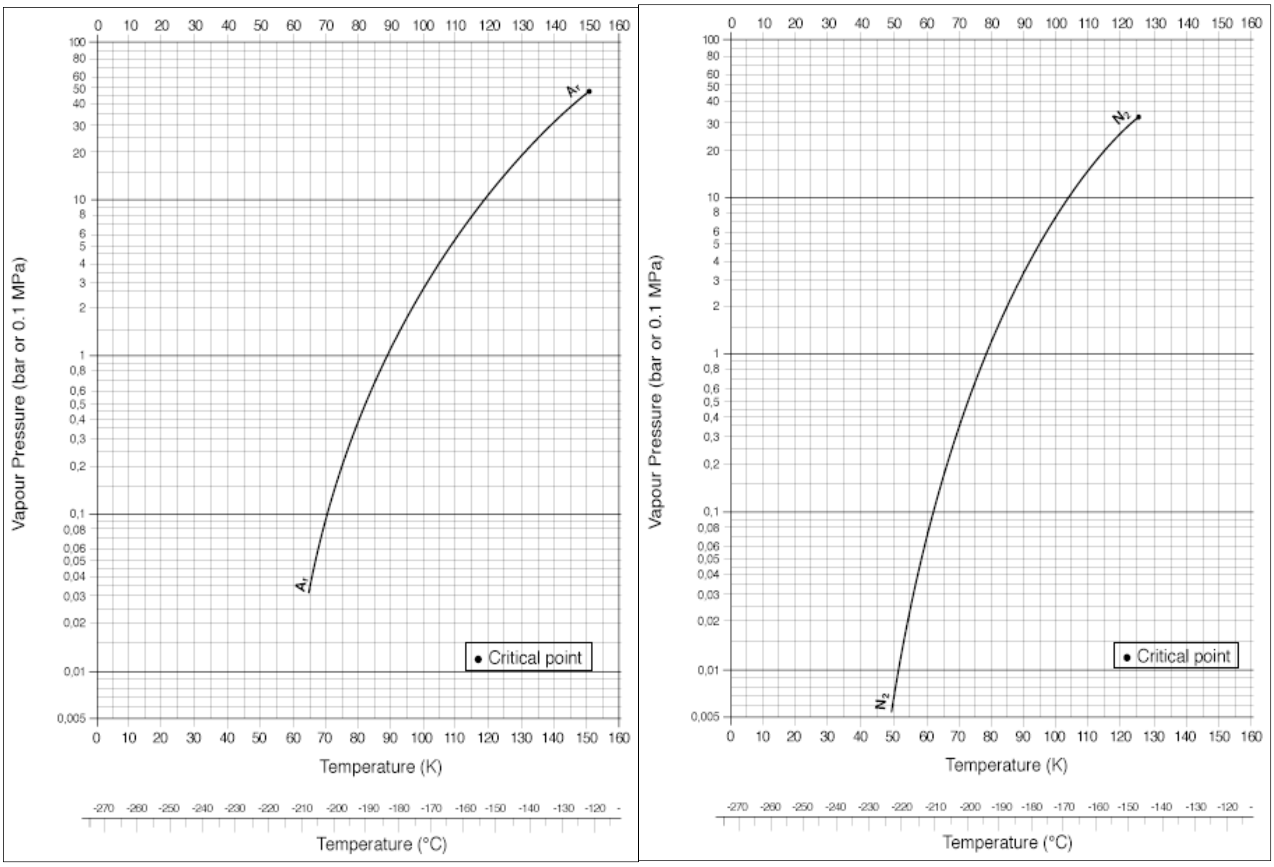


Figure 1 – Argon and Nitrogen vapor pressure curve

Component	**Pressure / Boiling Points**	
	1 bar	2 bar
Argon	87 K	95 K
Nitrogen	77 K	84 K

Table 1 – Boiling Points

A cryogenic distillation column performs a separation between gas and liquid by means of different boiling points and relative volatility of the components. The more volatile component will rise to the top of the column, while the less volatile one will be collected at the bottom.

The goal for our plant is to concentrate argon in the liquid phase at the bottom, and waste nitrogen in the gas phase at the to. More specifically, we will produce argon with very low nitrogen concentration from the bottom still.

We based the basic design of the distillation system on the McCabe-Thiele (M-T) method, one of the standard methods for the design and analysis of a distillation system. We first review the basic M-T method, to provide a first introduction to the matter, then we describe the apply it to our specific case.

Figure 2 illustrates the principle of the M-T method. The horizontal and vertical axes represent the concentration of the more volatile component in the liquid and gas phases. The thick solid curve is the equilibrium curve, and the thick solid lines are the condensation line and the collection line.

The main element in the distillation system is the column in which the gas-liquid equilibrium is maintained. The reboiler, at the bottom of the column, collects the liquid flowing down and boils part of the liquid using a heater.

The condenser, at the top of the column, plays a critical role in order to maintain a constant temperature profile along the column. The gas argon supplied at the input is cooled down to near boiling point, and then supplied to the feed point in the column at a flow rate F, as shown in the figure.

The argon processed through the column, with a lower nitrogen concentration than in the feed stock, is obtained from the reboiler, with flow rate W. The waste stream, argon with a higher nitrogen concentration, is collected from the top, with flow rate D.

In the following, the nitrogen concentration of each argon stream is expressed as x_F, x_W, x_D, respectively. The heating power of the reboiler coincides with the cooling power of the condenser at the top, and controls the flow of argon in the tower, indicated with L.

The reflux ratio, $R=L/D$, indicates the amount of argon returned to the column, compared with the amount extracted with a high concentration of nitrogen.

In the M-T method the distillation tower is assumed to be a connected series of theoretical stages, with the gas-liquid equilibrium changing by one step in each stage. The number of theoretical stages and the optimal position of the feed point are calculated for given boundary conditions of F, D, W, x_F, x_W, x_D, and R.

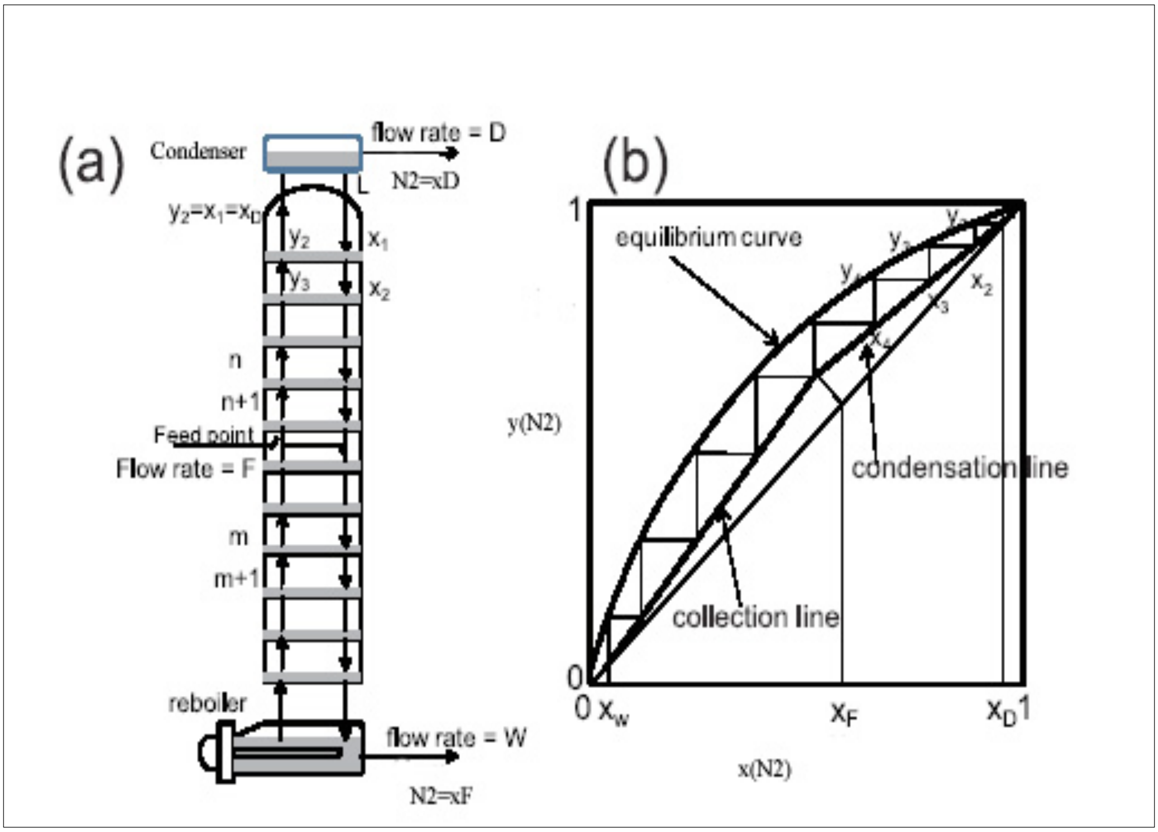


Figure 2 – McCabe-Thiele Method

In case of equilibrium between the liquid and gas phases in a binary mixture (here we refer specifically to the case of argon and nitrogen), Raoult's law relates the partial pressure of the gas phase elements, p_{Ar} and p_{N2}, to the fraction of each element in the liquid phase, x_{Ar} and x_{N2}:

$$p_{Ar} = P_{Ar} \cdot x_{Ar} \tag{1}$$

$$p_{N_2} = P_{N_2} \cdot x_{N_2} \tag{2}$$

$$P_{Ar} = 2\ bar \tag{3}$$

$$P_{N_2} = 5\ bar \tag{4}$$

where P_{Ar} and P_{N2} are the vapor pressure of argon and nitrogen at 95 K.

Raoult's law relates the concentration of nitrogen in the gas and liquid phase:

$$y_{N_2} = \frac{\alpha \cdot x_{N_2}}{1+(\alpha-1)\cdot x_{N_2}} \tag{5}$$

$$\alpha = \frac{P_{N_2}}{P_{Ar}} = 2.5 \tag{6}$$

Equation (5) is called the "Equilibrium curve", and it is the thick curve in Figure 2 (b)

For each theoretical stage, the conservation of mass flow relates the gas-phase and liquid-phase nitrogen concentrations in the neighboring stages:

$$y_{n+1} = \frac{R}{R+1} \cdot x_N + \frac{1}{R+1} \cdot x_D \tag{7}$$

$$y_{m+1} = \frac{R'}{R'-1} \cdot x_m + \frac{1}{R'-1} \cdot x_W \tag{8}$$

$$R' = \frac{L + q \cdot F}{W} \tag{9}$$

q is the fraction of liquid with respect to the total feed material.

Equation (7) is for the cells above the feeding point and represents the condensation line, whereas equation (8) is for the cells below the feeding point and represents the collection line. Figure 2 (b) shows the method to estimate the number of theoretical stages and the optimal feed point.

The following list summarizes the requirements for the distillation column:

1. The nitrogen concentration of the processed argon might be four orders of magnitude smaller than the original argon: $x_W = 10^{-6}$, $x_F = 10^{-2}$.
2. The collection efficiency of argon should be at or greater than 95%: $W/F = 0.95$, $D/F = 0.05$.
3. The system should be able to distill 10 Kg/day of inlet gas: $F = 0.417$ Kg/h.
4. The system should have a reflux ration of ~19: $R = 19$.
5. Argon is fed into the system in the liquid phase: $q = 1$.

Figure 3 shows the M-T diagram based on these requirements. The number of theoretical stages required is about 30, with the optimal inlet feed location at the 5th stage.

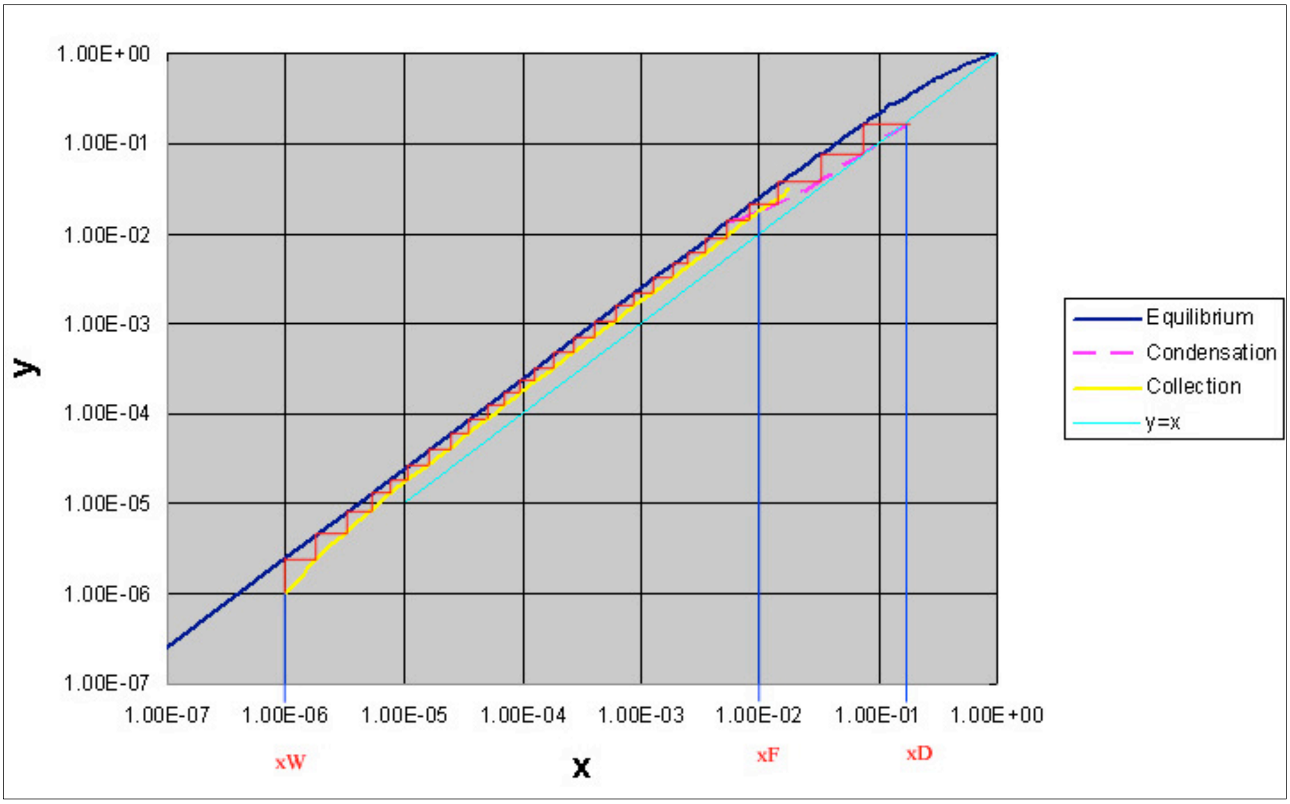


Figure 3 – M-T Diagram with system requirements

Experimental Apparatus

The packing inside the column is a key element of the distillation system. We will use the EX Laboratory Packing from Sulzer (see Figure 4) with a diameter of 0.834". The liquid load of this packing is between 0.48 $m^3/(m^2h)$ and 4.84 $m^3/(m^2h)$. For our application, the liquid load is 0.85 $m^3/(m^2h)$ for a reflux ratio $R \sim 19$.

The HETP for this packing is typically 5.3 cm for liquids (the specific value for argon was not immediately available from the company). The HETP depends strongly on the liquid load and on the type of liquid, so we conservatively increased this value by a factor two, thus the total overall packing length should be about 318 cm, equivalent to 60 stages. We will test this configuration and measure the real HETP; if the measured HETP is larger and more stages are needed, we will update the system to reach 1ppm of nitrogen concentration in the purified argon gas.

The optimal position of the feed was estimated to be the 5th stage from the top, 26.5 cm from the top.

The high pressure inside the gas bottle pushes the gas mixture into the cryogenic distillation plant. We plan to use a pump to drive the residual gas in the bottles, when its pressure falls below the pressure of the plant. The gas mixture, 99% argon, 1% nitrogen, will flow into the condensing volume, CV, will be liquefied, and then feed the column in the liquid phase.

The cooling power needed to chill down and liquefy the feed is provided through a temperature-controlled cryocooler.

Once the feed stock enters the column in the liquid phase, the argon-nitrogen mixture is purified by cryogenic distillation. An electric heater inside the reboiler will force the necessary boiling rate of the condensed argon to provide the desired reflux rate in the column. The reflux condenses in the condenser, and then flows to the reboiler through the column.

An electric heater on the output line permits to gasify the product and to store it in gas storage bottles. The cooling power needed to chill down the gas, the column, the piping, and to keep the desired temperature inside the system, is provided through a cryocooler.

The cryogenic distillation column, the reboiler, the condenser, and the cold piping, are insulated by a vacuum jacket and insulated with ten layers of super insulation, to reduce the heat loss for conduction and radiation.

Sampling lines are connected to a multi port Mass Spectrometer to measure argon and nitrogen contents of the inlet, the outlet, and the vent.

A specific set of temperature probes and heaters monitor and control the temperature inside the whole system. The whole system is equipped with a dedicated control system.

Summary

1) Design feed rate is ~10 Kg/day of feed stock, with the following concentrations:

Compound	Volumetric Concentration	Mass Concentration
Argon	99%	99.30%
Nitrogen	1%	0.70%
Inlet Flow rate		10 Kg/d

Table 2 – Inlet Concentrations

2) Possible Argon purification: ~99.9999%
3) Collection efficiency: 95%
4) Production rate: ~9.5 Kg/day

Compound	Volumetric Concentration	Mass Concentration
Argon	99.9999%	99.99993%
Nitrogen	0.0001%	0.000070%
Production rate		9.5 kg/d

Table 3 – Outlet Concentrations

5) To use commercially available parts, when possible, and equipments already available at Princeton University (Vacuum pumps, pump, booster)

Further Simulations

Process simulations have been performed in collaboration with Stevan Jovanovic from LINDE using UNISIM Simulation Software, an engineering suite widely used by oil and gas separation processes companies.

Compound	Volumetric Concentration	Mass Concentration
Argon	10%	13.70%
Nitrogen	90%	86.30%
Inlet Flow rate		10 kg/d

Table 4 – Inlet Concentrations

The design parameters were:

Variable	Value
Argon purification	1 ppm N2
Recovery	95 %
Expected HETP	75 mm
Inefficient packing	80 mm
Inter-bed high	50 mm

Table 5 – Design

Compound	Volumetric Concentration	Mass Concentration
Argon	99.9999%	99.99993%
Nitrogen	0.0001%	0.000070%
Production rate		9.5 kg/d

Table 6 – Outlet Concentrations

The simulation shows the middle of the packing as optimal position of the feed. Figure 5 and 6 show P&ID and PFD of the plant.

Figure 4 – Sulzer Laboratory Packing

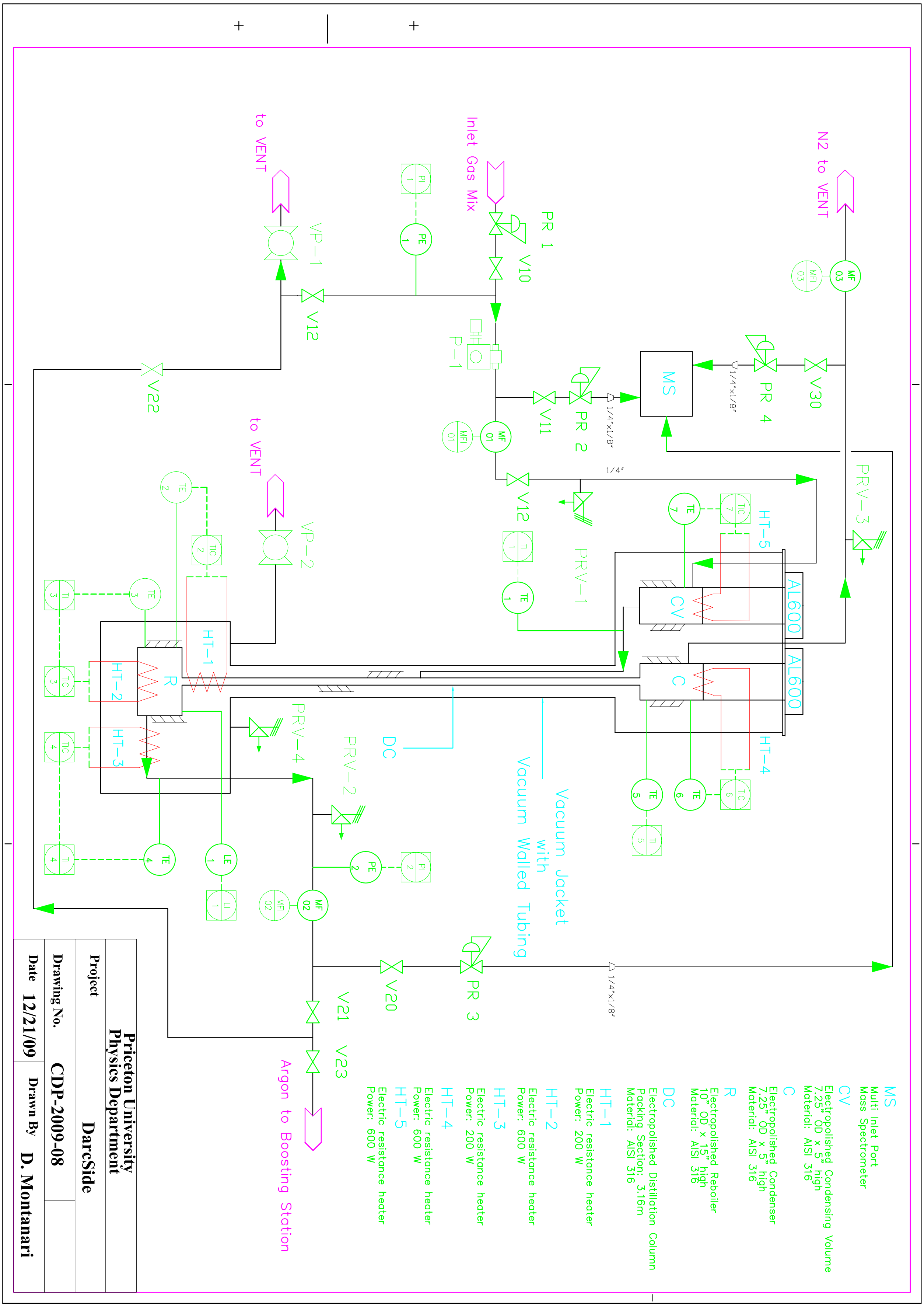
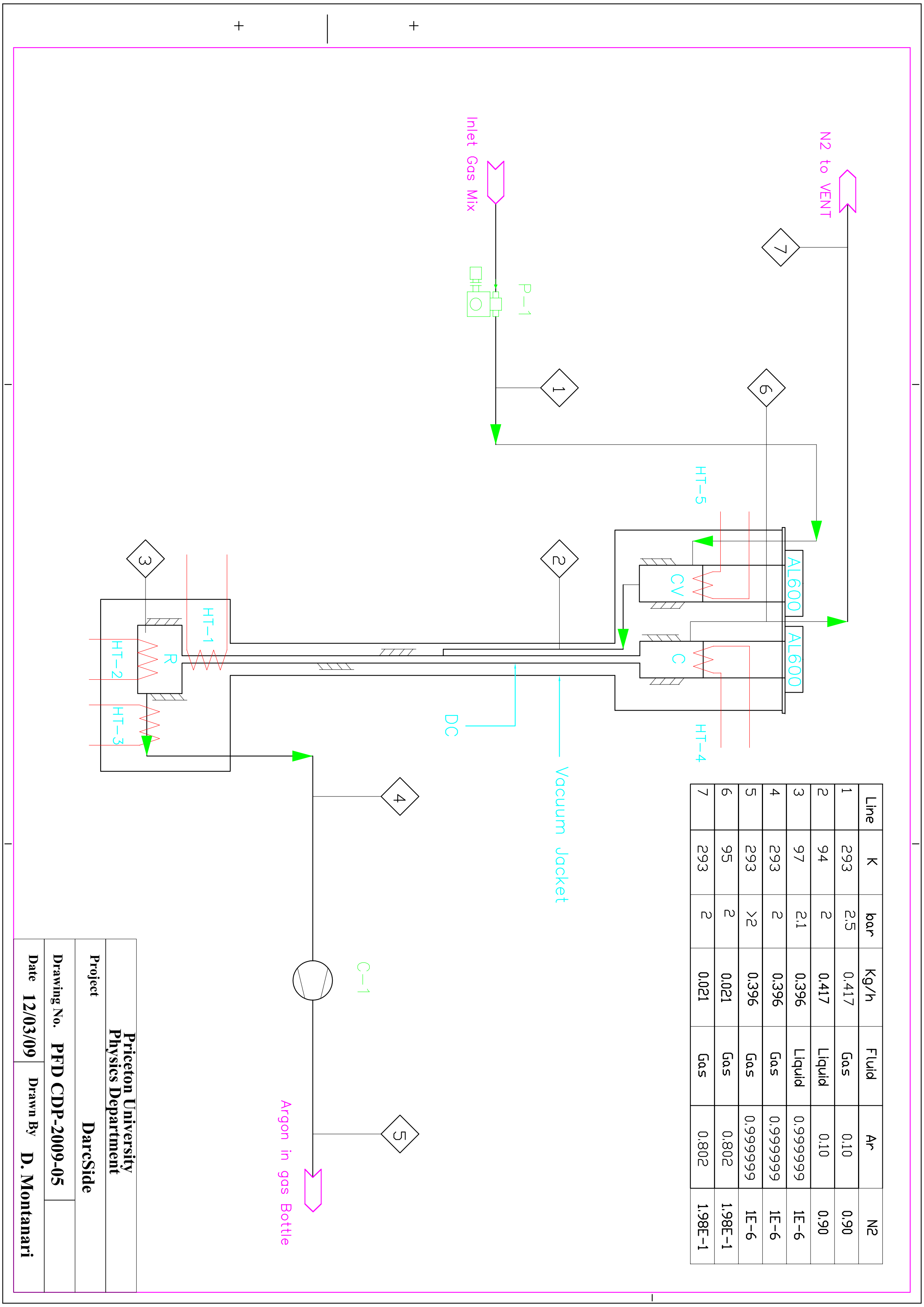


Figure 5 – P&ID of the plant



Line	K	bar	Kg/h	Fluid	Ar	N2
1	293	2.5	0.417	Gas	0.10	0.90
2	94	2	0.417	Liquid	0.10	0.90
3	97	2.1	0.396	Liquid	0.999999	1E-6
4	293	2	0.396	Gas	0.999999	1E-6
5	293	>2	0.396	Gas	0.999999	1E-6
6	95	2	0.021	Gas	0.802	1.98E-1
7	293	2	0.021	Gas	0.802	1.98E-1

Figure 6 – PFD of the Plant

Details on heat loss

The heat loss is mainly for conduction and radiation:

$$Q = \frac{K \cdot A \cdot \Delta T}{L} \tag{10}$$

$$\frac{\dot{Q}}{A} = \sigma \cdot \left(T_2^4 - T_1^4\right) \cdot \left[\frac{1}{e_1} + \frac{A_1}{A_2} \cdot \left(\frac{1}{e_2} - 1\right)\right]^{-1} \tag{11}$$

The vacuum jacket reduces the conduction heat transfer coefficient down to:

$$k_{Vacuum} = 5 \ \frac{mW}{K \cdot m} \tag{12}$$

The super insulation reduces both: the conduction heat transfer coefficient as follow,

$$k_{SI} = 10 - 50 \ \frac{\mu W}{K \cdot m} \tag{13}$$

Additionally, the heat loss for radiation by a factor 2^n, where *n* is the number of complete layers. Assuming 10 layers of super insulation rolled around the column, the piping, the reboiler and the condenser sides (not the top and the bottom flanges!), and assuming emissivity ~ 1, the total heat loss is of about 10 W.

The piping connected in and out the vacuum jacket will be welded with a vacuum jacket neck to reduce the heat loss for conduction in these connections.

The wires for the electrical sensors will be insulated to reduce both the joule effect and the conduction thermal effect to negligible values.

Details on cooling power

There are two independent cooling sources, used to chill down the inlet gas before it enters the column and liquefy the argon, and to keep the temperature profile along the column and liquefy the argon inside the column.

They are both two 600 W pulse tube cryocooler. The required cooling powers are:

$$Q_1 = m \cdot C_P \cdot \Delta T + \Delta H_V\left(Ar\right) + Q_{Lost} \tag{14}$$

$$Q_2 = m \cdot C_P \cdot \Delta T + \Delta H_V\left(Ar\right) + Q_{Lost} \tag{15}$$

$$Q_{Lost} = Q_{Rad} + Q_{Cond} + Q_{Contact} \tag{16}$$

where $Q_{Contact}$ is the cooling power lost for conduction in the various connection from outside at ambient temperature and inside, at the cryogenic temperature. $\Delta H_V\left(Ar\right)$ is the argon enthalpy of vaporization. The cooling power for the process is about 50+50 W.

Design Package

The system works under pressure, thus ASME Sect VIII Div 1 and 2 apply: condensing volume, condenser and reboiler are custom made, with ODs respectively of 7.25, 7.25 and 10 inches and a differential pressure across the wall of 30 psig; they do fall under the regulation.

The piping are standard 1 inch OD 0.083 inch wall and 0.25 inch OD 0.035 inch wall with ASME stamp. Valves and fittings are all from Swagelok and made according to ASME code.

In a thin cylindrical vessel under internal pressure, the radial stress, even though it is negative, is small compared to the hoop and longitudinal stress and can be assumed equal to zero; hence, either the maximum principal stress theory or the maximum shear stress theory give approximately the same results. We have chosen the first:

$$\sigma_{\max} \le \frac{\sigma_{YP}}{F_S} \tag{17}$$

$$F_S = 4 \tag{18}$$

$$\sigma_{YP} = 170MPa = 25ksi \tag{19}$$

$\sigma_{\max}$ is the maximum stress, σ_{YP} the yield strength and F_S the safety factor.

The system is working at cryogenic temperature, thus the yield strength will increase during the regular functioning of the plant; on the contrary, at the beginning we will bake out the system at 100C to outgas and remove impurities, nevertheless the yield strength value won't change.

The hoop and longitudinal stress in a cylindrical vessel subjected to internal pressure are respectively:

$$\sigma_1 = \frac{P \cdot r}{h} \tag{20}$$

$$\sigma_2 = \frac{P \cdot r}{2h} \tag{21}$$

$$\sigma_{\max} = \sigma_1 = \frac{P \cdot r}{h} \tag{23}$$

P is the internal pressure, *r* the internal radius and *h* the wall thickness.

The wall thickness becomes then:

$$h \ge F_S \cdot \frac{P \cdot r}{\sigma_{YP}} \tag{24}$$

$$h_{\mathrm{Re}b} \ge 0.022in \tag{25}$$

$$h_{Cond} = h_{Cond\_Vol} \ge 0.0168in \tag{26}$$

For the Reboiler, we have chosen a wall thickness of 0.25 inches, to which corresponds a maximum allowable internal working pressure of:

$$MAWP_{\mathrm{Re}b} = \frac{h \cdot \sigma_{YP}}{F_S \cdot r} = 316psi \tag{27}$$

For Condenser and Condensing Volume, we have chosen a wall thickness of 0.063 inches to which corresponds a maximum allowable internal working pressure of:

$$MAWP_{Cond} = \frac{h \cdot \sigma_{YP}}{F_S \cdot r} = 108\,psi \tag{28}$$

The maximum allowable working pressure of the plant is then 108 psi.

The vacuum jackets are under internal vacuum and at atmospheric pressure on the other side, thus the maximum differential pressure across the wall is 14.7 psig, less than the required 15 psig; they have been designed with a safety factor of 4.

If they loose the internal vacuum to outside, they will stabilize at atmospheric pressure, with no differential pressure across the wall; if the system leaks into the vacuum jacket, the internal pressure will stabilize at 14.7 psig. Since they are double wall vacuum jacket (see Fig. 7), the differential pressure across the wall will be 30 psig, thus this case has to be analyzed as pressurized vessels. Eq. 17 to 19 are still valid:

$$\sigma_{\max} \le \frac{\sigma_{YP}}{F_S} \tag{17}$$

$$F_S = 4 \tag{18}$$

$$\sigma_{YP} = 170\,MPa = 25\,ksi \tag{19}$$

$\sigma_{\max}$ is the maximum stress, σ_{YP} the yield strength and F_S the safety factor.

The maximum stress in a cylindrical vessel subjected to internal pressure is expressed by (20):

$$\sigma_{\max} = \sigma_1 = \frac{P \cdot r}{h} \tag{20}$$

The wall thickness becomes then equation (24):

$$h \ge F_S \cdot \frac{P \cdot r}{\sigma_{YP}} \tag{24}$$

$$h_{Top} \ge 0.056\,in \tag{29}$$

$$h_{Middle} \ge 0.024\,in \tag{30}$$

$$h_{Bottom} \ge 0.046\,in \tag{31}$$

To uniform the vacuum jackets to the two already in our hands from Linde, we have chosen the same wall thickness of 0.125 inches, to which correspond maximum allowable working pressures of:

$$MAWP_{Top} = \frac{h \cdot \sigma_{YP}}{F_S \cdot r} = 65\,psi \tag{32}$$

$$MAWP_{Middle} = \frac{h \cdot \sigma_{YP}}{F_S \cdot r} = 145\,psi \tag{33}$$

$$MAWP_{Top} = \frac{h \cdot \sigma_{YP}}{F_S \cdot r} = 78\,psi \tag{34}$$

The maximum allowable working pressure of the vacuum jackets is then 65psi.

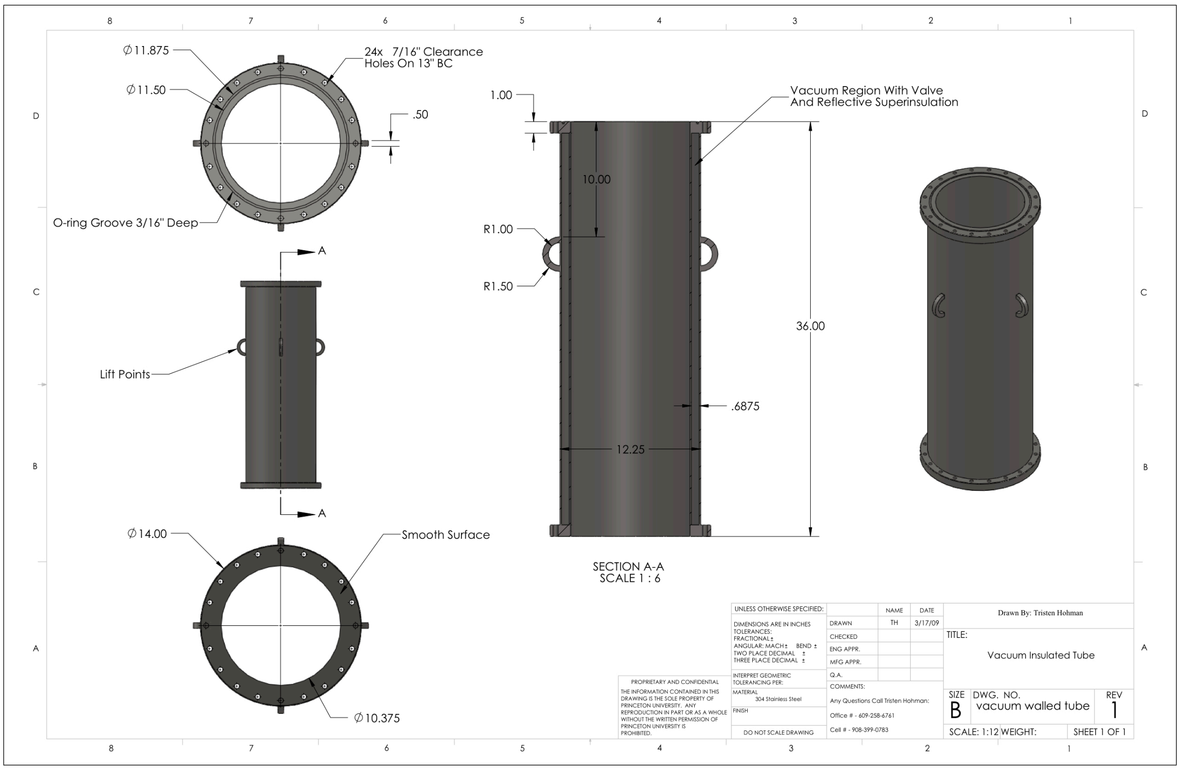


Figure 7 - Double Wall Vacuum jacket

Failure Mode Analysis

There are two possible failure mode scenarios:

1) loss of vacuum in the vacuum jacket
2) loss of cooling power (AL-600 malfunctioning or power cut)

They can both result in a sudden increase of temperature, thus pressure, inside the system. A combination of the two, although very unlikely to happen, represents the worst case scenario. Since it is not possible to isolate with valves or similar devices any part of the system from inlet to outlet and vent, we can consider one whole system and design a single pressure relieving safety system.

The Reboiler is the most critical part of the system: it contains liquid argon that can boil up and increase the pressure inside the system, as a consequence of loss of vacuum and/or cooling power.

According to ASME Sect VIII Div. 1 and 2, a relief system has been design following these principles:

$$MHWP_{Plant} = 108\,psi \tag{28}$$

$$P_{Set} < 1.10 \cdot MHWP \tag{35}$$

The wetted area is defined by A_{wet}:

$$A_{wet} = \pi \cdot D \cdot \left(\frac{D}{4} + E \right) = 2.1\ sq\ ft \tag{36}$$

$$D = 9.75\ in \tag{37}$$

$$E = 7.5\ in \tag{38}$$

D is the Reboiler diameter and *E* the effective liquid level.

The total heat absorption is defined by *Q*:

$$Q = 34{,}500 \cdot F \cdot A_{wet}^{0.82} = 63{,}393 \ {}^{BTU}\!/_{hr} \tag{39}$$

$$F = 1 \tag{40}$$

F is the environmental factor, equal to one in case of a bare vessel W/O insulation (vacuum in this case).

The fluid mass flow converted to gas from the liquid, *W*, is :

$$W = {}^{Q}\!/_{H_{vap}} = 912 \ {}^{lb}\!/_{hr} \tag{41}$$

$$H_{vap} = 69.5 \ {}^{BTU}\!/_{lb} \tag{42}$$

H_{vap} is the latent heat of vaporization.

The minimum required relieving area is defined by:

$$A = \frac{W \cdot \sqrt{T \cdot Z}}{C \cdot K \cdot P_1 \cdot K_b \cdot \sqrt{M}} \tag{43}$$

$$T = 110K = 198R \tag{44}$$

$$Z = 1 \tag{45}$$

$$P_1 = P_{set} \cdot 1.16 + 14.7 = 82 psia \tag{46}$$

$$P_{set} = 58 psig \tag{47}$$

$$C = 377 \tag{48}$$

$$K = 0.975 \tag{49}$$

$$K_b = 1 \tag{50}$$

$$M = 39.95 \tag{51}$$

T is the temperature of the fluid at the valve inlet, *Z* the compressibility factor (conservatively taken as unit for gas or vapor), P_1 the relieving pressure, P_{set} the set pressure, *C* the C-value, *K* the effective coefficient of discharge, K_b the capacity correction factor due to back pressure (takes as unit for atmospheric back pressure and back pressure above relieving pressure ratio below 0.55) and *M* the molecular weight. When sizing for multiple pressure relief valves, the total required area is calculated on an overpressure of 16% (Eq. 46).

The minimum required relieving area is:

$$A = 0.067 \ sq \ in \tag{43}$$

A BP series or 900 series from Crosby, with an orifice of 0.074 square inch, would be required to relieve the flow caused by loss of cooling power and loss of vacuum on this application. Although we don't have a single line with that cross section available (Eq. 53), it is possible to combine together inlet, outlet and vent, placing a pressure safety relief valve on each line:

$$OD = 0.25 \ in \tag{52}$$

$$A_{pipe} = 0.0254 \ sq \ in \tag{53}$$

$$A_{Tot} = 3 \cdot A_{pipe} = 0.076 \ sq \ in \tag{54}$$

The sound level at 100 feet from the discharge point is express in decibel by:

$$L_{100} = L + 10 \cdot Log_{10}\left(0.29354 \cdot W_{Max} \cdot k \cdot \frac{T}{M}\right) = 96dB \tag{55}$$

$$L = 57 \tag{56}$$

$$k = 1.66 \tag{57}$$

$$W_{Max} = \frac{A_{Select} \cdot P_1 \cdot C \cdot K \cdot K_b \cdot \sqrt{M}}{\sqrt{T \cdot Z}} = 3{,}006 \ {}^{lb}\!/_{hr} \tag{58}$$

$$A_{Select} = 3 \cdot 0.074 = 0.222 \ sq \ in \tag{59}$$

L is the noise intensity (from graph), *W* the maximum relieving capacity based on the selected valve, whose area is A_{Select}, *k* the ratio of specific heats of the fluid.

There is another failure scenario: the combination of the one just analyzed, loss of vacuum in the vacuum jacket and contemporary loss of cooling power in the system, with leak from the system to the vacuum jacket.

It this happens we will pressurize the vacuum jacket up to 58 psig, pressure at which the pressure relief system of the plant will start relieving the inside pressure outside (Eq. 47).

$$MHWP_{VJ} = 65 psia \tag{32}$$

The minimum required relieving area is defined by:

$$A = \frac{W \cdot \sqrt{T \cdot Z}}{C \cdot K \cdot P_1 \cdot K_b \cdot \sqrt{M}} \tag{43}$$

$$W = 912 \, {}^{lb}\!/_{hr} \tag{41}$$

$$T = 110K = 198R \tag{44}$$

$$Z = 1 \tag{45}$$

$$P_1 = P_{set} \cdot 1.10 + 14.7 = 64.2 psia \tag{60}$$

$$P_{set} = 45 psig \tag{61}$$

$$C = 377 \tag{48}$$

$$K = 0.975 \tag{49}$$

$$K_b = 1 \tag{50}$$

$$M = 39.95 \tag{51}$$

W is the mass flow rate of the gas flowing from inside to the vacuum jacket, *T* the temperature of the fluid at the valve inlet, *Z* the compressibility factor (conservatively taken as unit for gas or vapor), P_1 the relieving pressure, P_{set} the set pressure, *C* the C-value, *K* the effective coefficient of discharge, K_b the capacity correction factor due to back pressure (taken as unit for atmospheric back pressure and back pressure above relieving pressure ratio below 0.55) and *M* the molecular weight.

The minimum required relieving area is:

$$A = 0.086 \ sq \ in \tag{62}$$

A type D BP series from Crosby with an orifice of 0.11 square inches may be a good solution; anyway, since during normal operations the vacuum jacket is under vacuum, it might be better to go with a vacuum tight rupture disk that will allow to pull vacuum inside and keep the system leak tight.

There are several 2.75" ports on the vacuum jacket that are suitable for this with a relieving area of 0.679 square inches.

The sound level at 100 feet from the discharge point is express in decibel by:

$$L_{100} = L + 10 \cdot Log_{10}\left(0.29354 \cdot W_{Max} \cdot k \cdot \frac{T}{M}\right) = 91dB \tag{55}$$

$$L = 56 \tag{56}$$

$$k = 1.66 \tag{57}$$

$$W_{Max} = \frac{A_{Select} \cdot P_1 \cdot C \cdot K \cdot K_b \cdot \sqrt{M}}{\sqrt{T \cdot Z}} = 1{,}200 \ {}^{lb}\!/_{hr} \tag{63}$$

$$A_{Select} = 0.11 \ sq \ in \tag{64}$$

L is the noise intensity (from graph), *W* the maximum relieving capacity based on the selected valve, whose area is A_{Select}, *k* the ratio of specific heats of the fluid.

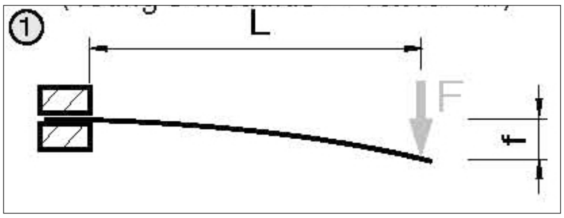
Framing

A support structure for the cryogenic distillation has been designed (Fig. 11-12) and already mounted in the high bay at Princeton University, Physics Dept. (Fig. 13-14).

The overall weight of the distillation plant is 2,050 lbs, with the center of mass located 57" below the top flange of the top vacuum jacket, almost on the 1" column.

The critical points of the structure are:

1) Vertical Beams 157" high with gusseted bottom: the buckling load is 15,388 lbs
2) Vertical Beams to support the bottom 32" high with gusseted bottom: the buckling load is 33,100 lbs
3) Top Horizontal Braces: 1x24" long, 4x23" long with 1170 lbs of total load. The center of mass is located in the middle of the top section, we can then assume that the load is well distributed on 5x24" braces.



The most stressed section is the support section:

the load is $$F = \frac{1170}{5} = 234 \ lbs \tag{65}$$

the length is $$l = 24 \ in \tag{66}$$

the stress is $$\sigma = \frac{F \cdot l}{Z_x} = 3{,}330 \ {}^{lbs}\!/_{in^2} \tag{67}$$

the torque (moment) is $$M = F \cdot l = 5{,}620 \ lbs \cdot in \tag{68}$$

the shear force is $$V = F = 234 \ lbs \tag{69}$$

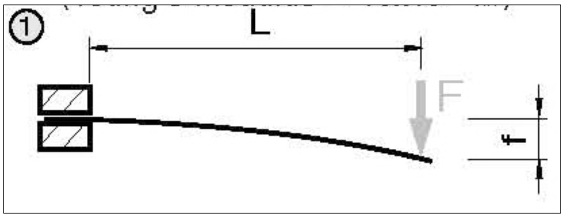
the shear stress is $$\tau = \frac{V \cdot Z_x}{I_x \cdot t} = 40 \ {}^{lbs}\!/_{in^2} \tag{70}$$

the total stress is $$\sigma_{Tot} = \sqrt{\sigma^2 + 3 \cdot \tau^2} = 3{,}340 \ {}^{lbs}\!/_{in^2} \tag{71}$$

$$\sigma_{Max} = 28{,}000 \ {}^{lbs}\!/_{in^2} \tag{72}$$

the deflection is	$f = 0.035" \ (0.9\%)$	(73)
the static moment of area are	$Z_x = 1.690\ in^3, Z_y = 0.889\ in^3$	(74)
the moment of inertia are	$I_x = 2.995\ in^4, I_y = 0.787\ in^4$	(75)

4) Bottom Horizontal Braces: 4x20" long with 880 lbs of total load. The center of mass is located in the middle of the bottom section, we can then assume that the load is well distributed on 4x20" braces.



The most stressed section is the support section:

the load is	$F = 220\ lbs$	(76)
the length is	$l = 20\ in$	(77)
the stress is	$\sigma = \frac{F \cdot l}{Z_x} = 2{,}610\ {}^{lbs}\!/_{in^2}$	(78)
the torque (moment) is	$M = F \cdot l = 4{,}400\ lbs \cdot in$	(79)
the shear force is	$V = F = 220\ lbs$	(80)
the shear stress is	$\tau = \frac{V \cdot Z_x}{I_x \cdot t} = 36\ {}^{lbs}\!/_{in^2}$	(81)
the total stress is	$\sigma_{Tot} = \sqrt{\sigma^2 + 3 \cdot \tau^2} = 2{,}620\ {}^{lbs}\!/_{in^2}$	(82)
	$\sigma_{Max} = 28{,}000\ {}^{lbs}\!/_{in^2}$	(72)
the deflection is	$f = 0.019" \ (0.5\%)$	(83)

The static moment of area and the moment of inertia are shown in Eq. 74-75.

5) 10mm T-bolt: maximum vertical load capacity 2,698 *lbs* each

Mechanical Drawings

Figure 8, 9 and 10 show mechanical drawings of the plant: assembly, top and bottom view respectively. The support framing has been already mounted and the design is shown in Figure 11 and 12; Figure 13 and 14 show the framing as already assembled at Princeton University.

Manpower and Equipment

Manpower and equipment required from FNAL to complete the project are:

1) Safety review: ½ week engineer

2) Procedure review and completion: ½ weeks engineer
3) Assembly: 3 weeks engineer, 3 weeks technician, 1 week programmer
4) Commissioning: 3 weeks engineer, 3 weeks technician
5) Purification of 150 kg or argon: 3 weeks, plant running 24 hours, need one single shift with 1 technician and 1 engineer
6) Operations on the field, Cortez, CO, to free Eng. David Montanari: 1 technician for 2 months
7) 1 Laptop/desktop to run the control system ($2,000)
8) 2 Multi port Mass Spectrometer or gas Analyzer (2 x $60,000 = $120,000). One of the two units for use in FNAL and the second unit to upgrade the unit currently in Cortez.
9) Leak tight compressor/booster to pressurize argon into the storage bottle from 30 psia up to 4,200 psia, at a flow rate of 10-20 kg/d ($15,000)
10) Twenty-five (25) high pressure gas storage cylinders, with maximum pressure: 4,600 psi ($15,000)

Conclusions

We designed a cryogenic distillation plant capable to reduce nitrogen concentration in the feedstock by four orders of magnitude, reaching a concentration of 10^{-6} N_2/Ar, starting from a feed concentration of nitrogen from argon of 10^{-2} N_2/Ar. The collection efficiency is 95% and the process flow rate is about 0.417 Kg/h of gas.

The flow rate of the argon with low nitrogen concentration, collected from the bottom, is about 0.396 Kg/h, that corresponds to 9.5 Kg/h. The flow rate of the argon with high nitrogen concentration, vented from the top, is about 0.021 Kg/h.

Part List

N	Material	Supplier	Note
1	Resin for cryogenic temperature	Swagelok	ALREADY AVAILABLE
1	Low Temperature Conductivity Epoxy	Lakeshore	ALREADY AVAILABLE
1	Staycast Epoxy 2850-FT, Catalyst 9	Lakeshore	ALREADY AVAILABLE
1	Apiezion Grease		ALREADY AVAILABLE
1	Vacuum Grease		ALREADY AVAILABLE
3	Capillary lines 1/16"	SRS	**To be procured (FNAL)**
1	Multiport Mass Spectrometer	SRS	**To be procured (FNAL)**
10 ft	EP stainless steel tubing 1/8" – sampling lines	Swagelok or other	**To be procured (FNAL)**
40 ft	EP stainless steel tubing ¼" – process lines	Swagelok	ALREADY AVAILABLE
6	Silicon Diode temperature sensors	Lakeshore	ALREADY AVAILABLE
1	Control System National Instruments	NI	ALREADY AVAILABLE
1	Desktop/Laptop to run the control system	t.b.d.	**To be procured (FNAL)**
1	Labview License	Labview	ALREADY AVAILABLE
8	¼" Solenoid EP High Flow Diaphragm Valves	Swagelok	ALREADY AVAILABLE
4	¼" Needle Valves Sampling Lines	Swagelok	ALREADY AVAILABLE
1	Pressure Regulator (from 220 to 4 bar)	Swagelok	ALREADY AVAILABLE
3	In Line Pressure Regulator (4 to <1 bar)	Swagelok	ALREADY AVAILABLE
3	Mass Flow Controllers	Sierra Instruments	ALREADY AVAILABLE
1	Vacuum Pump – Vacuum Jacket	Varian	ALREADY AVAILABLE
1	Leak Detector - High Vacuum	???	ALREADY AVAILABLE
1	Vacuum Pump - Initial Cleaning	Varian	ALREADY AVAILABLE
1	Vacuum Pump - Filling the bottle	Varian	ALREADY AVAILABLE

1	Recirculation Pump - Start Up (15-20 l/m)	???	ALREADY AVAILABLE
2	Pressure Transducers	MKS	ALREADY AVAILABLE
1	LAr Level Sensor & Controller 13.77" Active length	American Magnetics	ALREADY AVAILABLE
1	Reboiler (10" OD x 15" h)	Custom	ALREADY AVAILABLE
1	Condensing Volume (7.25" OD x 5" h)	Custom	ALREADY AVAILABLE
1	Condenser (7.25" OD x 5" h)	Custom	ALREADY AVAILABLE
1	Distillation Column (1" OD)	Custom	ALREADY AVAILABLE
60st	Packing – 60 Stages	Sulzer	ALREADY AVAILABLE
2	Electric heater blocks with cartridges 0.25" and 1" OD	Janis	ALREADY AVAILABLE
1	Electric heater block - Bottom Reboiler	Janis	ALREADY AVAILABLE
3	Power Supplies for heaters	Agilent	ALREADY AVAILABLE
1	Flexible Hose 20" KH-025-VCR-20	Key High Vacuum	ALREADY AVAILABLE
1	Flexible Hose 15" KH-025-VCR-15	Key High Vacuum	ALREADY AVAILABLE
3	VJ – Column	Custom	ALREADY AVAILABLE
1	VJ – Bottom	Custom	ALREADY AVAILABLE
1	VJ – Top	Custom	ALREADY AVAILABLE
2	Stainless steel Flanges - Top VJ	Custom	ALREADY AVAILABLE
1	Stainless steel Flange – Lid Bottom VJ	Custom	ALREADY AVAILABLE
1	Stainless steel Flange - Lid Reboiler	Custom	ALREADY AVAILABLE
1	Copper Flange – Bottom Reboiler	Custom	ALREADY AVAILABLE
10	Copper Gaskets for Conflat Flanges (various sizes)	Kurt Lesker	ALREADY AVAILABLE
1	Cryogenic O-Ring for Copper Flange	Creavey	ALREADY AVAILABLE
1	Superinsulation	t.b.d.	**To be procured (FNAL)**
2	AL 600 cryocooler fully equipped	Cryomech	ALREADY AVAILABLE
2	Air Cooled Helium Compressor for AL 600	Cryomech	ALREADY AVAILABLE
2	Set of special tools for mounting AL 600	Cryomech	ALREADY AVAILABLE
2	Set of lines for AL 600	Cryomech	ALREADY AVAILABLE
2	Heater blocks for AL 600	Janis	ALREADY AVAILABLE
2	Temperature Controller for AL 600 Mod 336	Lakeshore	ALREADY AVAILABLE
2	Fluid feed through ¼"	Kurt Lesker	ALREADY AVAILABLE
8	9 PIN feed through	AccuGlass	ALREADY AVAILABLE
1	Relief Valve SS-RL3S4	Swagelok	ALREADY AVAILABLE
2	Argon and Nitrogen gas bottles for testing	t.b.d.	ALREADY AVAILABLE
1	Empty Bottle to mix the gas	t.b.d.	ALREADY AVAILABLE
	Fittings from the bottles to the line	t.b.d.	ALREADY AVAILABLE
3	Adapters ¼" VCR - MNPT	Swagelok	ALREADY AVAILABLE
10	¼" EP Butt weld "Tee"	Swagelok	ALREADY AVAILABLE
20	VCR Female nuts DI	Swagelok	ALREADY AVAILABLE
20	VCR gaskets with retainer for high flow diaphragm valves	Swagelok	ALREADY AVAILABLE
20	EP VCR short glands	Swagelok	ALREADY AVAILABLE
1	KF Connector for Vacuum Pump	Key High Vacuum	ALREADY AVAILABLE
1	Support framing structure – Custom made	Bosch RexRoth	ALREADY AVAILABLE
	Coaxial cables, pin feed-through, connectors	Accuglass	ALREADY AVAILABLE
	Screws, Nuts, Bolts for flanges	Kurt Lesker	ALREADY AVAILABLE

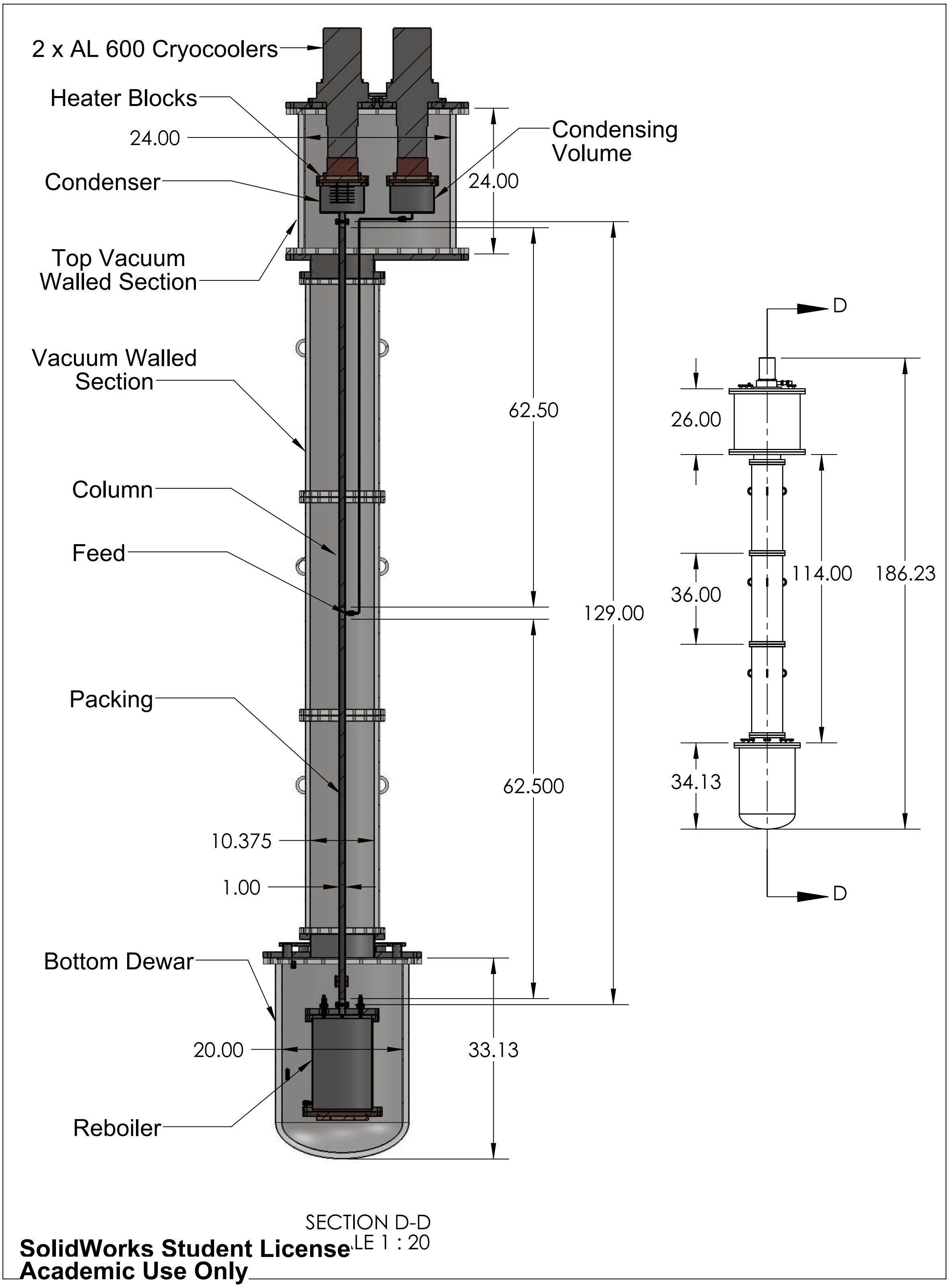


Figure 8 – Assembly

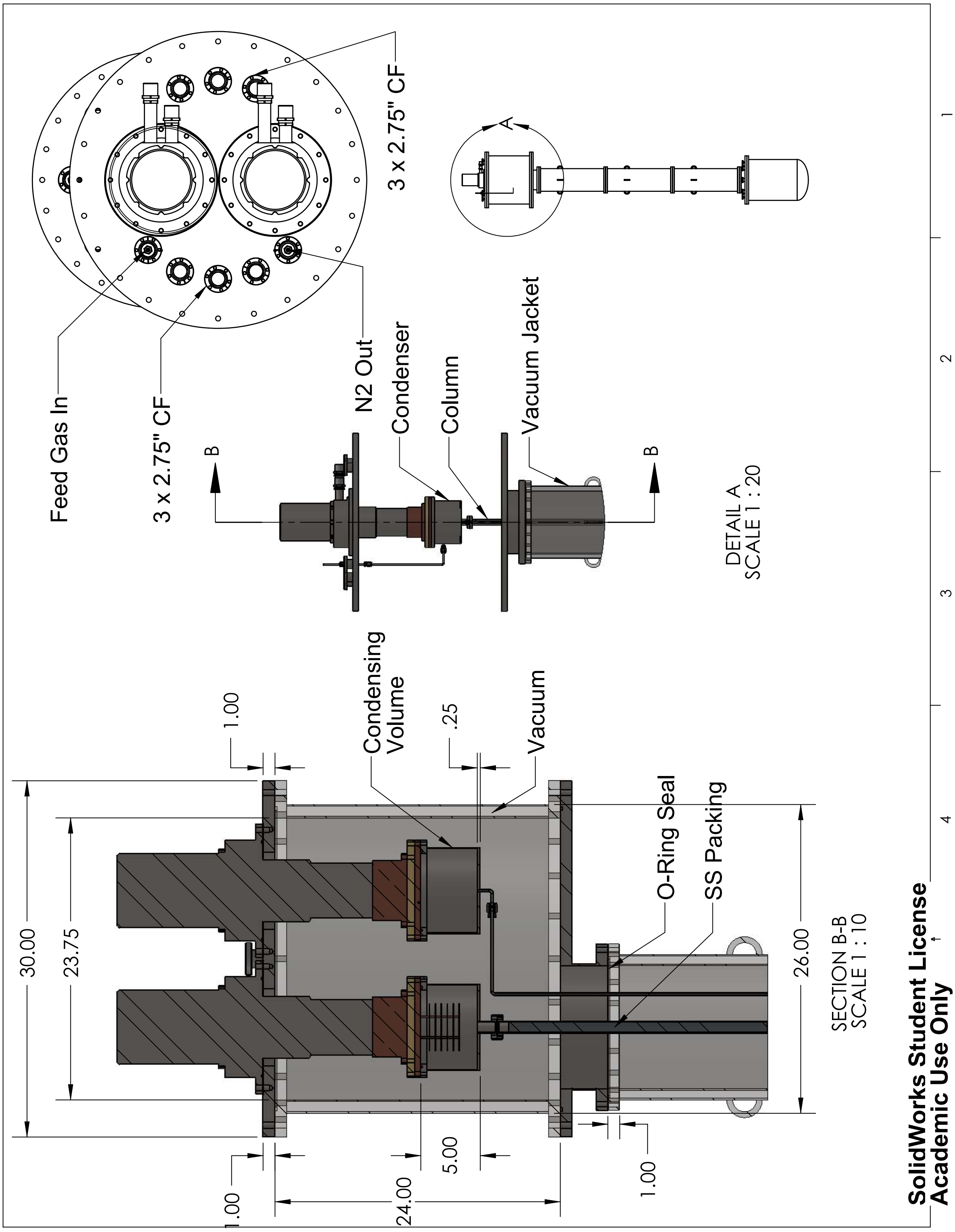


Figure 9 – Top view

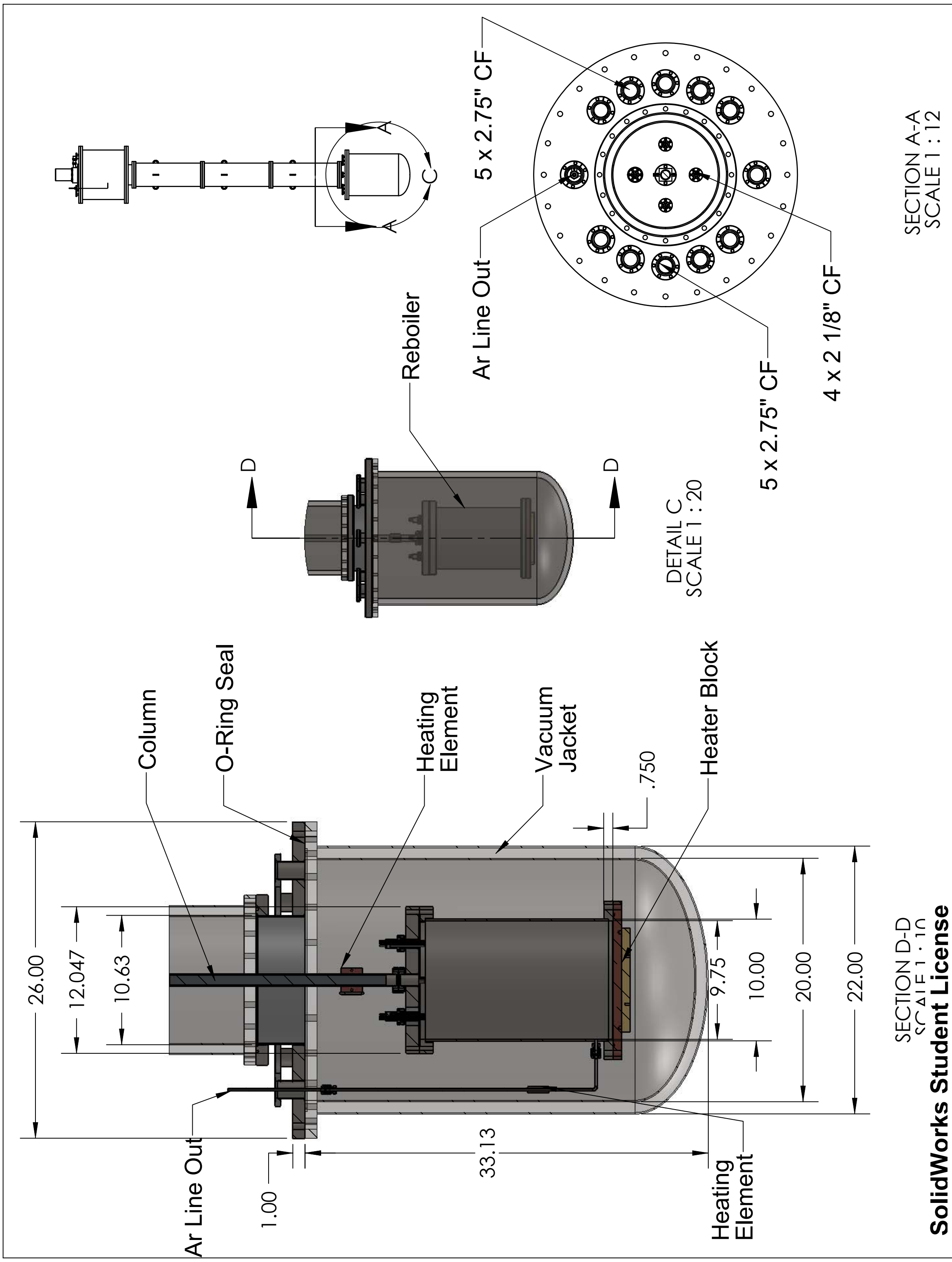


Figure 10 – Bottom view

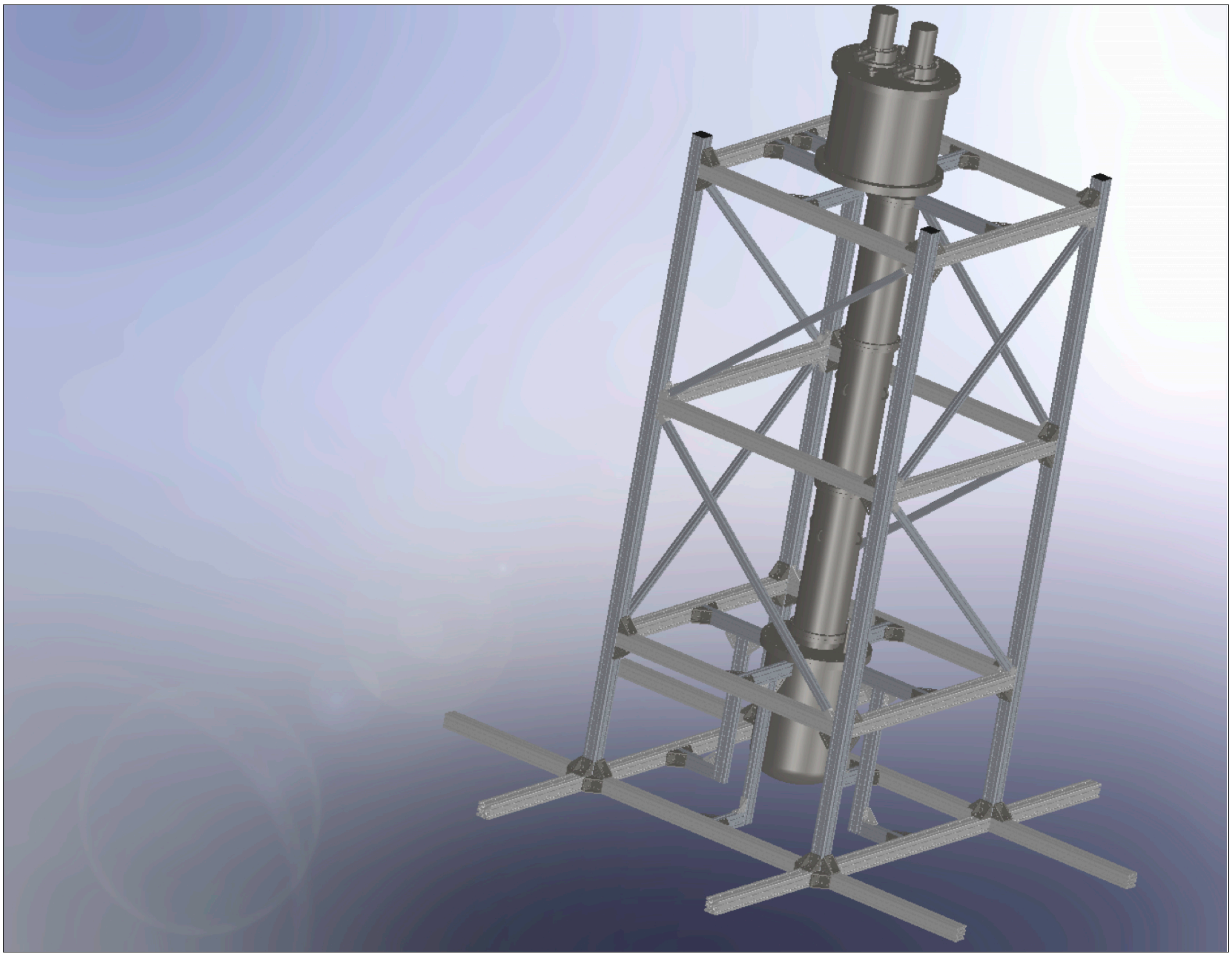


Figure 11 – Support Framing with distillation column

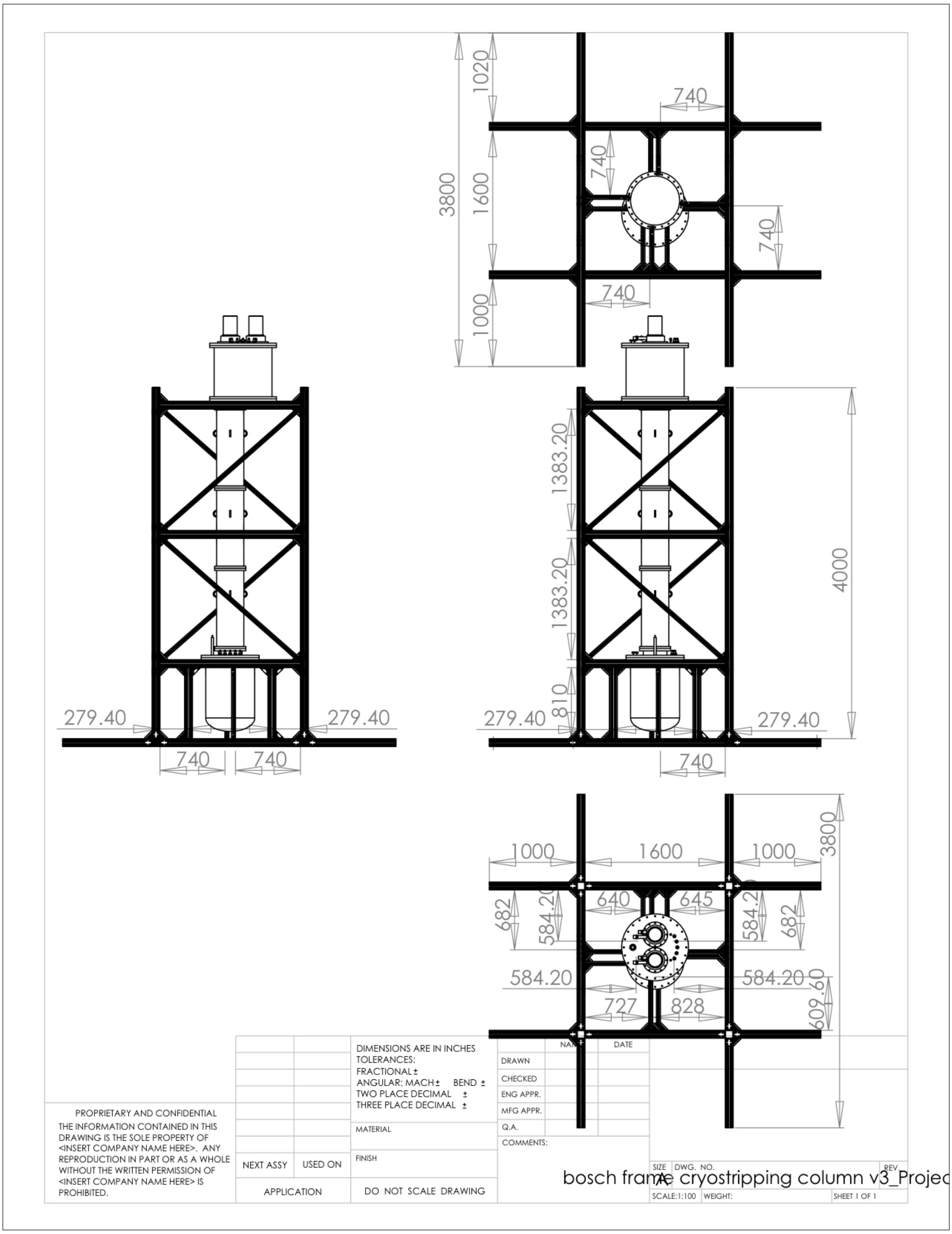


Figure 12 – Support Framing with distillation column

Figure 13 - Support structure at Princeton University

Figure 14 - Support structure with mounting crew at Princeton University